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(54) STICK COMPOSITIONS AND A PROCESS FOR THEIR MANUFACTURE

(71) We, SCHERING AKTINGE-SELLSCHAFT, a Body Corporate organised according to the laws of Germany, of Berlin and Bergkamen, Germany, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The present invention is concerned with stick compositions, especially medicament-containing sticks suitable for topical application, and with a process for their manufacture.

Medicament-containing sticks for topical application and cosmetic sticks having anti-perspirant and deodorizing properties have been known. Such sticks may be produced on the basis of alcoholic soap gels or solid hydrogels using alginates, pectins or carboxyl-vinyl polymers. In German Offenlegungsschrift No. 1,617,472 there are described stick compositions made of polyalkylene glycol ethers of fatty alcohols containing 12 to 22 carbon atoms and polyalkylene glycol esters of wax acids.

There have also been known medicament-containing lubricating sticks that are made with paraffins and/or cocoa butter, in some cases with the addition of alkali soaps.

All the known stick compositions are of little use for the production of medicament-containing sticks that are sufficiently lubricating and capable of being removed by washing, and cosmetically unobjectionable. Furthermore, sticks based on alcoholic soap gels have the disadvantages of deficient shape-stability and incompatibility towards various medicinally active substances. These stick compositions are removable by washing, but they have no lubricating activity. Sticks based on paraffin and/or cocoa butter do possess the desired lubricating activity, but these stick compositions however cannot easily be removed by washing and are always somewhat sticky, so that they do not slide sufficiently over surfaces to which they are applied.

There has accordingly been a need for com-

position for a medicament-containing stick that is an inert carrier for medicinally active substance and has both a lubricating and an oil-restoring, emollient activity, but does not impart an oily shine to the skin and can easily be removed by washing with water.

The present invention is based on the observation that this need is met by a medicament or cosmetic base formed by mixing together a solid wax, a fatty alcohol, a polyhydroxylated monoester of glycerine and, if desired, a higher saturated hydrocarbon and/or a fatty acid alkanolamide.

The present invention accordingly provides a composition which comprises a solid wax, a fatty alcohol and a polyhydroxylated monoester of glycerine and, if desired, a higher hydrocarbon and/or a fatty acid alkanolamide, especially such a composition which also contains a medicament and is in the form of a stick suitable for topical application or such a composition which is in the form of a cosmetic stick suitable for topical application.

The present invention also provides a process for the manufacture of a stick composition, wherein a solid wax, a fatty alcohol and a polyhydroxylated monoester of glycerine and also, if desired, a higher hydrocarbon and/or a fatty acid alkanolamide are mixed together; an important variant of the process of the present invention comprises mixing the aforesaid substances and medicament together and forming sticks from the resulting mixture. The term "higher hydrocarbon" is used herein to mean a hydrocarbon having a viscosity of at least 50 cP at 20°C.

The present invention therefore includes a process for the manufacture of a medicament-containing stick, wherein a solid wax, a fatty alcohol, a medicament and a polyhydroxylated monoester of glycerine and, if desired, a higher hydrocarbon (as hereinbefore defined) and/or a fatty acid alkanolamide are mixed together.

As solid waxes there may be mentioned all those that are toxicologically unobjectionable and are suitable for the manufacture of stick

[Price 33p]

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 Exhibit 8

compositions. They have a melting point of at least 50°C but preferably not higher than 90°C. Solid waxes which may be employed for example beeswax, carnauba wax or spermaceti or other waxes having similar physical properties.

As fatty alcohols there may be mentioned alcohols containing 8 to 30 carbon atoms, for example myristic alcohol, cetyl alcohol and stearyl alcohol and similar higher fatty alcohols.

The polyhydroxylated monoester of glycerine used in accordance with the present invention is, more especially, polyhydroxylated monoester of glycerine derived from an alcohol or alcohols containing up to 5 carbon atoms and from a carboxylic acid containing 8 to 18 carbon atoms and preferably having a molecular weight within the range of from 200 to 600 inclusive; advantageously there may be used a mixture of such esters, for example, bis-polyethoxylene glyceryl monostearate and monomyristate. The polyhydroxylated monoester of glycerine or mixture of esters may be used in an amount of 15 to 70% by weight inclusive, calculated on the total weight of the composition.

The medicaments used in accordance with the present invention are preferably, for example, corticoids, for example 11 β ,17 α ,21-trihydroxy- Δ^4 -pregnene-3,20-dione (hydrocortisone) and esters thereof, 11 β ,17 α ,21-trihydroxy- $\Delta^1,4$ -pregnadiene-3,20-dione (prednisolone) and esters thereof, 6 α -fluoro-11 β ,21-dihydroxy - 16 α - methyl- $\Delta^1,4$ -pregnadiene-3,20-dione (fluocortolone) and esters thereof, 6 α ,9-d-fluoro - 11 β ,21-dihydroxy-3,20-dioxo-16 α -methyl- $\Delta^1,4$ -pregnadiene (difluocortolone) and esters thereof, 6 α -fluoro-9,11 β -dichloro-21-hydroxy - 16 α - methyl- $\Delta^1,4$ -pregnadiene-3,20-dione and esters thereof, 6 α ,11 β -difluoro-9 - chloro - 21 - hydroxy - 16 α - methyl- $\Delta^1,4$ -pregnadiene-3,20-dione and esters thereof, 9 α - chloro-6 α - fluoro - 11 β ,21 - dihydroxy-16 α - methyl - $\Delta^1,4$ -pregnadiene-3,20-dione (clorocortolone) and esters thereof, 6-chloro-17 α - hydroxy - 1 α ,2 α - methylene - Δ^4 -pregnadiene-3,20-dione (cyproterone) and esters thereof, and also 9-fluoro-11 β ,17,21-trihydroxy - 3,20-dioxo - 16 α - methyl- $\Delta^1,4$ -pregnadiene (dexamethasone) and esters thereof, and/or bactericides of the type of 5-amino - 1,3 - bis(ethylhexyl) - hexahydro-5-methyl-pyrimidine (hexetidine), 5,7-dichloro-8-hydroxy-quinaldine (chloroquinaldol) and benzalkonium chloride.

There are also suitable as medicaments, however, local anaesthetics, antiseptics and disinfectants, for example para-aminobenzoic acid ethyl ester (anaesthin), ω -diethylamino-2,6-dimethyl-acetanilide (xylocaine) and its hydrochloride, or a polyethylene oxide ether of dedecyl alcohol (thesit), or boric acid, 1-hexadecyl-pyridinium chloride (cetyl-

pyridinium chloride), surfen, anthrasol, ichthylol or tumenol, or cignolin or hexachlorophene. The terms "xylocaine", "thesit", "surfen", "ichthylol", "tumenol" and "cignolin" are Registered Trade Marks.

As higher hydrocarbons, that are optionally added, there are to be understood, more especially, saturated hydrocarbons, and in particular the various types of paraffin, for example hard, that is solid, paraffin, viscous paraffin and thinly liquid paraffin, with a viscosity of at least 50 cP at 20°C.

As fatty acid alkanolamides there are preferred those derived from lower alcohols containing 1 to 5 carbon atoms, for example methanol or ethanol, and from higher fatty acids containing 8 to 25 carbon atoms, for example myristic acid.

If desired, there may be incorporated in the compositions other substances that are inert towards medicaments, for example perfume oils and/or colouring agents.

The incorporation of the medicaments that may be used in the stick compositions of the invention is advantageously effected by dissolving the medicament directly in a melt of the composition. If the medicament does not dissolve or dissolves incompletely in this melt, it is very finely triturated with a part or the whole of the cold liquid constituents of the mixture. A melt of the remainder of the constituents of the mixture is then added to this suspension, the method of preparation of the composition not being critical provided that it is capable of being poured while molten into the moulds used to form the sticks.

To make the stick compositions of the present invention the still liquid composition is advantageously poured into suitable screw-type extensible cases with a core as the packing material (stick casings).

The stick compositions of the present invention, for example the medicament-containing sticks, have the advantages of being shape-stable and of having a good appearance. They are tolerated by the skin, as they contain no tenside. At the same time they have a lubricating and an emollient activity without imparting a shininess to the skin. The stick compositions slide easily over the skin and can easily be washed off with water.

The proportions of the components employed in the stick compositions of the present invention are generally:

	Percentage by Weight	
Solid wax	10—70	
Higher fatty alcohol	20—35	
Polyhydroxylated mono- ester(s) of glycerin	15—70	
Higher hydrocarbon	0—25	125
Fatty acid alkanolamide	0—40	
Medicament or cosmetic agent	0.005—20	

Especially preferred are sticks containing a medicament or cosmetic agent dissolved in a solid mixture comprising 4—15% by weight of cetyl or stearyl alcohol, 10—25% by weight of beeswax and 50—70% by weight of the polyhydroxylated monoester of glycerine mixture employed in the Examples below, alone or as a mixture with up to equal parts by weight of a paraffin or microcrystalline wax.

The following Examples illustrate the invention. Instead of incorporating medicinally active substances as described in some of the Examples, the compositions may equally well have other additives introduced into them by mixing, so that such stick compositions can also be used for making, for example, cosmetic preparations, for example lipsticks, deodorants, eyeshadows and perfume sticks. The mixture of polyhydroxylated monoesters of glycerine used was CETIOL HE (Henkel & Cie, Düsseldorf). "CETIOL" is a Registered Trade Mark.

Example 1

5.0 Grams of cetyl alcohol, 25.0 grams of beeswax, 20.0 grams of spermaceti, 17.2 grams of paraffin oil and 32.8 grams of a mixture of polyhydroxylated monoesters of glycerine were melted together. The melt was poured into stick cases.

Example 2

10.0 Grams of cetyl alcohol, 25.0 grams of beeswax and 65.0 grams of a mixture of polyhydroxylated monoesters of glycerine were melted together. The melt was poured into stick cases.

Example 3

12.50 Grams of beeswax, 18.75 grams of coconut fatty acid monoethanolamide, 12.50 grams of stearyl alcohol, 10.50 grams of viscous paraffin and 43.75 grams of mixture of polyhydroxylated monoesters of glycerine were melted together. The melt was poured into stick cases.

Example 4

7.5 Grams of cetyl alcohol, 25.0 grams of beeswax, 10.0 grams of a microcrystalline wax synthesized from branched chain hydrocarbons, 15.0 grams of viscous paraffin and 42.5 grams of a mixture of polyhydroxylated monoesters of glycerine were melted together. The melt was poured into stick cases.

Example 5

5.0 Grams of cetyl alcohol, 25.0 grams of beeswax, 20.0 grams of hard paraffin (melting at 51—53°C), 17.2 grams of viscous paraffin and 32.8 grams of a mixture of polyhydroxylated monoesters of glycerine were melted together. The melt was poured into stick cases.

Example 6

0.025 Gram of fluocortolone trimethylacetate and 0.05 gram of chloroquinaldol were dissolved in a melt of 3.185 grams of a mixture of polyhydroxylated monoesters of glycerine, 0.490 gram of cetyl alcohol and 1.225 grams of bleached wax with the addition of 0.025 gram of Crematest perfume oil, and the whole was poured into stick cases. "Crematest" is a Registered Trade Mark.

Example 7

0.05 Gram of chloroquinaldol were dissolved in a melt of 3.185 grams of a mixture of polyhydroxylated monoesters of glycerine, 0.490 gram of cetyl alcohol and 1.25 grams of beeswax with the addition of 0.025 gram of Crematest perfume oil, and the whole was poured into stick cases.

WHAT WE CLAIM IS:—

1. A composition which comprises a solid wax, a fatty alcohol and a polyhydroxylated monoester of glycerine.
2. A composition as claimed in claim 1, containing a higher hydrocarbon (as hereinbefore defined).
3. A composition as claimed in claim 1 or 2, containing a fatty acid alkanolamide.
4. A composition as claimed in any one of claims 1 to 3, wherein the solid wax is beeswax.
5. A composition as claimed in any one of claims 1 to 3, wherein the solid wax is spermaceti.
6. A composition as claimed in any one of claims 1 to 5, wherein the fatty alcohol is myristic alcohol.
7. A composition as claimed in any one of claims 1 to 5, wherein the fatty alcohol is cetyl alcohol.
8. A composition as claimed in any one of claims 1 to 5, wherein the fatty alcohol is stearyl alcohol.
9. A composition as claimed in any one of claims 1 to 8, wherein the polyhydroxylated monoester of glycerine is derived from an alcohol or alcohols containing up to 5 carbon atoms and from a carboxylic acid containing 8 to 18 carbon atoms.
10. A composition as claimed in claim 9, wherein the polyhydroxylated monoester of glycerine has a molecular weight of from 200 to 600.
11. A composition as claimed in any one of claims 1 to 10, which contains a mixture of two or more polyhydroxylated monoesters of glycerine.
12. A composition as claimed in claim 11, which contains a mixture of bis-polyethoxylene-glyceryl-mono-stearate and -mono-myristate.
13. A composition as claimed in any one of claims 1 to 12, wherein the polyhydroxylated monoester(s) of glycerine is or are present in

- a total amount within the range of from 15 to 70% by weight inclusive, calculated on the weight of the composition.
- 5 14. A composition as claimed in any one of claims 1 to 13, containing a perfume oil and/or a colouring agent.
- 15 15. A composition as claimed in any one of claims 1 to 14, containing a medicament.
- 10 16. A composition as claimed in claim 15, wherein the medicament is a corticoid and/or a bactericide.
17. A composition as claimed in claim 15, wherein the medicament is a local anaesthetic, an antiseptic or a disinfectant.
- 15 18. A composition as claimed in any one of claims 15 to 17, which is in the form of a stick suitable for topical application.
19. A composition as claimed in any one of claims 1 to 14, which is in the form of a cosmetic preparation.
- 20 20. A composition as claimed in claim 19, which is in the form of a stick suitable for topical application.
- 25 21. A composition as claimed in claim 1, having a composition substantially as described in any one of Examples 1 to 7 herein.
22. A process for the manufacture of a composition as claimed in claim 1, wherein a solid wax, a fatty alcohol and a polyhydroxylated monoester of glycerine are mixed together and, if desired, formed into a stick.
- 30 23. A process as claimed in claim 22, wherein a solid wax, a fatty alcohol, a polyhydroxylated monoester of glycerine and a higher hydrocarbon (as hereinbefore defined) and/or a fatty acid alkanolamide are mixed together.
- 35 24. A process as claimed in claim 22 or 23, wherein the solid wax is beeswax.
- 40 25. A process as claimed in claim 22 or 23, wherein the solid wax is spermaceti.
26. A process as claimed in any one of claims 22 to 25, wherein the fatty alcohol is myristic alcohol.
- 45 27. A process as claimed in any one of claims 22 to 25, wherein the fatty alcohol is cetyl alcohol.
28. A process as claimed in any one of claims 22 to 25, wherein the fatty alcohol is stearyl alcohol.
- 50 29. A process as claimed in any one of claims 22 to 28, wherein the polyhydroxylated monoester of glycerine is derived from an alcohol or alcohols containing up to 5 carbon atoms and from a carboxylic acid containing 8 to 18 carbon atoms.
- 55 30. A process as claimed in claim 29, wherein the polyhydroxylated monoester of glycerine has a molecular weight of from 200 to 600.
- 60 31. A process as claimed in any one of claims 22 to 30, wherein there is used a mixture of two or more polyhydroxylated monoesters of glycerine.
- 65 32. A process as claimed in claim 31, wherein a mixture of bis-polyethoxylene-glyceryl-mono-stearate and -mono-myristate is used.
33. A process as claimed in any one of claims 22 to 32, wherein the polyhydroxylated monoester(s) of glycerine is or are used in a total amount within the range of from 15 to 70% by weight inclusive, calculated on the weight of the composition.
- 70 34. A process as claimed in claim 22, conducted substantially as described in any one of Examples 1 to 5 herein.
- 75 35. A process for the manufacture of a medicament-containing stick, wherein a solid wax, a fatty alcohol, a medicament and a polyhydroxylated monoester of glycerine are mixed together.
- 80 36. A process as claimed in claim 35, wherein a solid wax, a fatty alcohol, a medicament, a polyhydroxylated monoester of glycerine, and a higher hydrocarbon (as hereinbefore defined) and/or a fatty acid alkanolamide are mixed together.
- 85 37. A process as claimed in claim 35 or 36, wherein the solid wax is beeswax.
- 90 38. A process as claimed in claim 35 or 36, wherein the solid wax is spermaceti.
39. A process as claimed in any one of claims 35 to 38, wherein the fatty alcohol is myristic alcohol.
- 95 40. A process as claimed in any one of claims 35 to 38, wherein the fatty alcohol is cetyl alcohol.
41. A process as claimed in any one of claims 35 to 38, wherein the fatty alcohol is stearyl alcohol.
- 100 42. A process as claimed in any one of claims 35 to 41, wherein the medicament is a corticoid and/or a bactericide.
43. A process as claimed in any one of claims 35 to 41, wherein the medicament is a local anaesthetic, an antiseptic or a disinfectant.
- 105 44. A process as claimed in any one of claims 35 to 41, wherein the polyhydroxylated monoester of glycerine is derived from an alcohol or alcohols containing up to 5 carbon atoms and from a carboxylic acid containing 8 to 18 carbon atoms.
- 110 45. A process as claimed in claim 44, wherein the polyhydroxylated monoester of glycerine has a molecular weight of from 200 to 600.
- 115 46. A process as claimed in any one of claims 35 to 45, wherein there is used a mixture of two or more polyhydroxylated monoesters of glycerine.
- 120 47. A process as claimed in claim 46, wherein a mixture of bis-polyethoxylene-glyceryl-mono-stearate and -mono-myristate is used.
- 125 48. A process as claimed in any one of claims 35 to 47, wherein the polyhydroxylated monoester(s) of glycerine is or are used in a total amount within the range of from 15 to 130

70% by weight inclusive, calculated on the weight of the medicament-containing stick.

49. A process as claimed in claim 48, conducted substantially as described in Example

5 6 or 7 herein.

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